A LEVEL CHEMISTRY

TOPIC 20 – CHROMATOGRAPHY AND SPECTROSCOPY

TEST

Answer all questions

Max 50 marks

Name ……..
Mark ……../50 ……....% Grade ……

1. A tripeptide was heated with hydrochloric acid and a mixture of amino acids was formed. This mixture was separated by column chromatography.
Outline briefly why chromatography is able to separate a mixture of compounds.
Practical details are **not** required.

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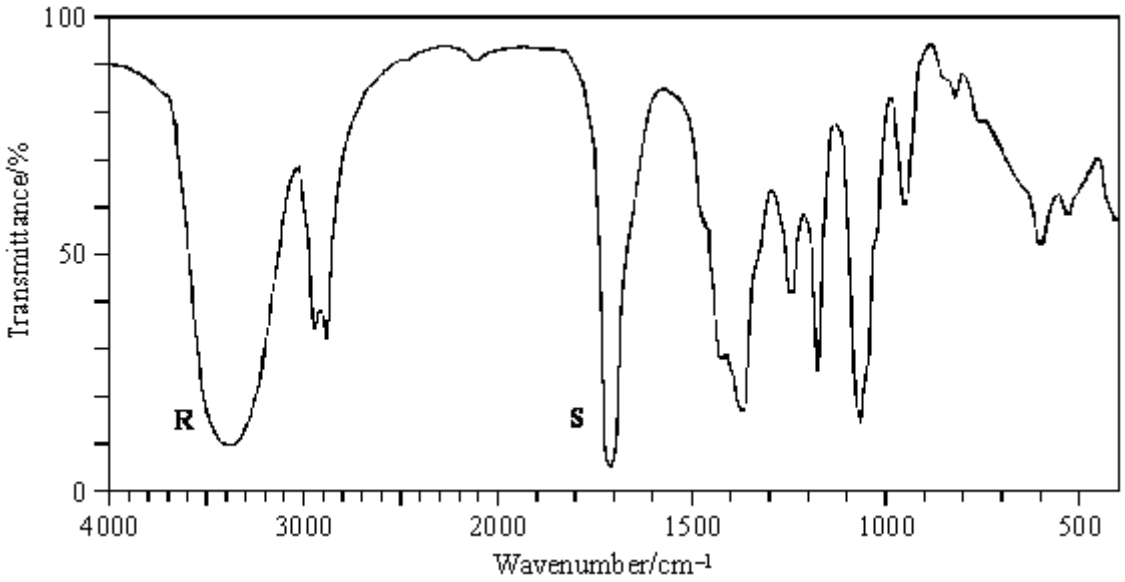
(3)
(Total 3 marks)

2. Spectral data for use in this question are provided below the Periodic Table (first item on the database).

Compound **Q** has the molecular formula $C_4H_8O_2$

(a) The infra-red spectrum of **Q** is shown below.



Identify the type of bond causing the absorption labelled **R** and that causing the absorption labelled **S**.

R .....

S .....

(2)

(b) **Q** does not react with Tollens' reagent or Fehling's solution. Identify a functional group which would react with these reagents and therefore cannot be present in **Q**.

.....

(1)

(c) Proton n.m.r. spectra are recorded using a solution of a substance to which tetramethylsilane (TMS) has been added.

(i) Give two reasons why TMS is a suitable standard.

Reason 1 .....

Reason 2 .....

(ii) Give an example of a solvent which is suitable for use in recording an n.m.r. spectrum. Give a reason for your choice.

Solvent .....

Reason .....

(4)

(d) The proton n.m.r. spectrum of **Q** shows 4 peaks.

The table below gives δ values for each of these peaks together with their splitting patterns and integration values.

δ/ppm	2.20	2.69	3.40	3.84
Splitting pattern	singlet	triplet	singlet	triplet
Integration value	3	2	1	2

What can be deduced about the structure of **Q** from the presence of the following in its n.m.r. spectrum?

(i) The singlet peak at δ = 2.20

.....

(ii) The singlet peak at δ = 3.40

.....

(iii) Two triplet peaks

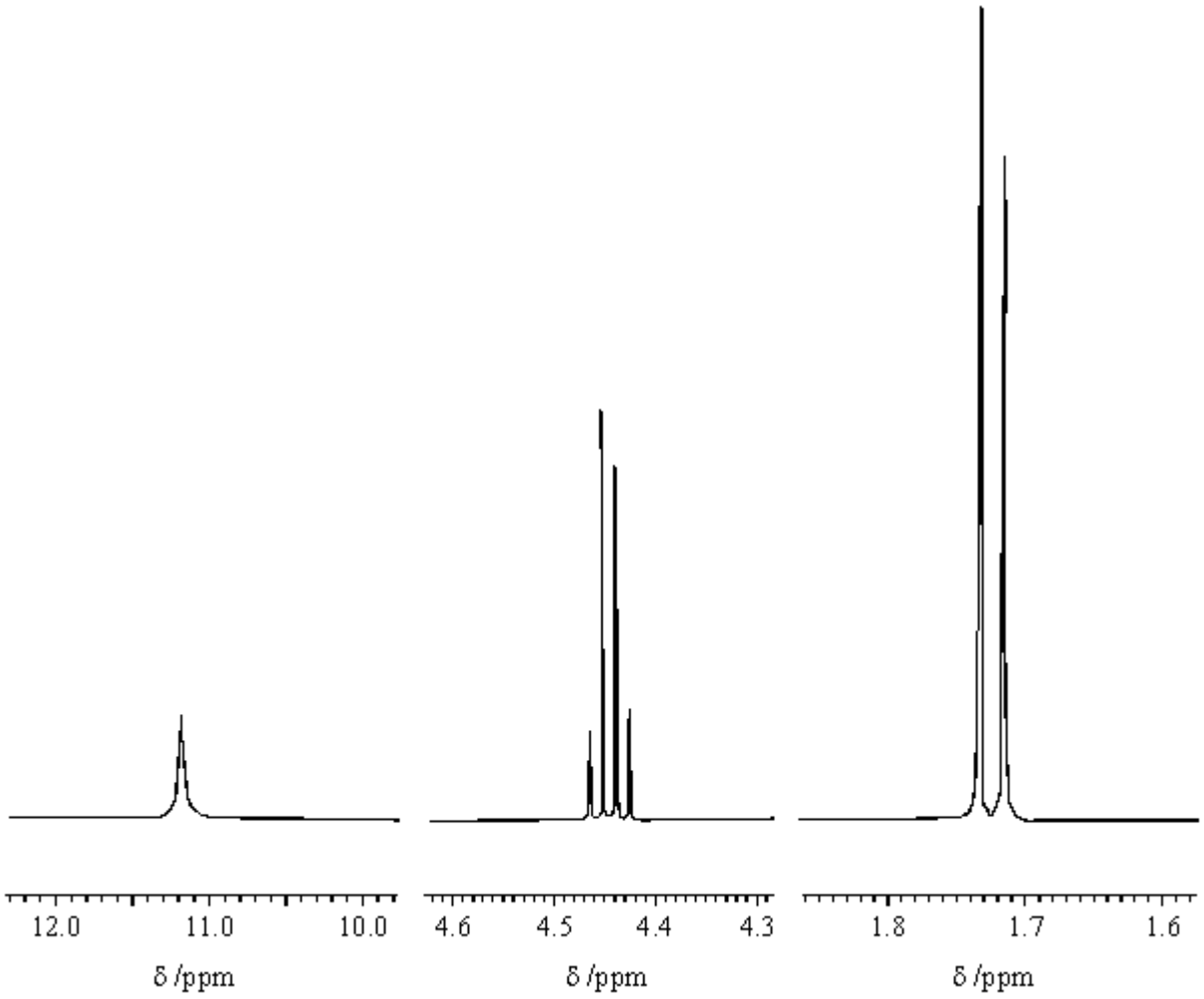
.....

(3)

(e) Using your answers to parts (a), (b) and (d), deduce the structure of compound **Q**.

(1)
(Total 11 marks)

3. Three sections of the proton n.m.r. spectrum of $CH_3CHClCOOH$ are shown below.



(a) Name the compound $CH_3CHClCOOH$

.....

(1)

(b) Explain the splitting patterns in the peaks at δ 1.72 and δ 4.44

.....

.....

.....

(2)

(c) Predict the splitting pattern that would be seen in the proton n.m.r. spectrum of the isomeric compound $ClCH_2CH_2COOH$

.....

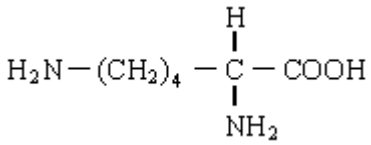
.....

(1)

(d) The amino acid *alanine* is formed by the reaction of $CH_3CHClCOOH$ with an excess of ammonia. The mechanism is nucleophilic substitution. Outline this mechanism, showing clearly the structure of *alanine*.

(5)

(e) The amino acid *lysine* has the structure



Draw structures to show the product formed in each case when lysine reacts with

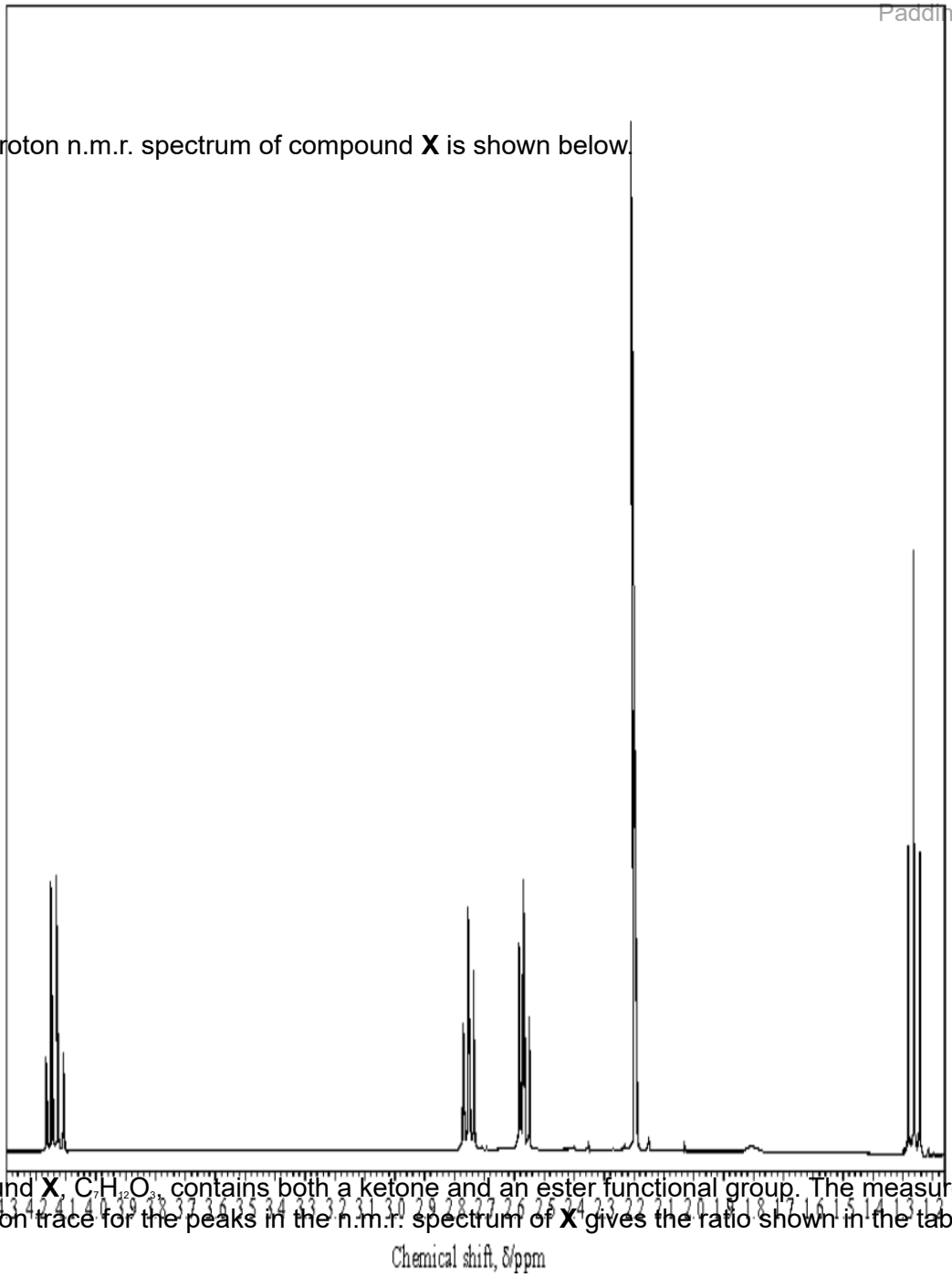
(i) an excess of aqueous HCl,

(ii) an excess of aqueous NaOH,

(iii) another molecule of lysine.

(3)
(Total 12 marks)

4. The proton n.m.r. spectrum of compound **X** is shown below.



Compound **X**, $C_7H_{12}O_3$, contains both a ketone and an ester functional group. The measured integration trace for the peaks in the n.m.r. spectrum of **X** gives the ratio shown in the table below.

Chemical shift, δ/ppm	4.13	2.76	2.57	2.20	1.26
Integration ratio	0.8	0.8	0.8	1.2	1.2

Refer to the spectrum, the information given above and the data below the Periodic Table provided to answer the following questions.

(a) How many different types of proton are present in compound **X**?

.....

(1)

(b) What is the whole-number ratio of each type of proton in compound **X**?

.....

(c) Draw the part of the structure of **X** which can be deduced from the presence of the peak at δ2.20.

.....

(1)

(d) The peaks at δ4.13 and δ1.26 arise from the presence of an alkyl group. Identify the group and explain the splitting pattern.

Alkyl group .....

Explanation .....

.....

.....

(3)

(e) Draw the part of the structure of **X** which can be deduced from the splitting of the peaks at δ2.76 and δ2.57.

.....

(1)

(f) Deduce the structure of compound **X**.

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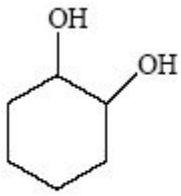
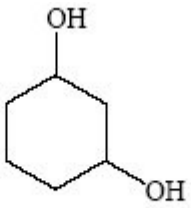
(2)

(Total 9 marks)

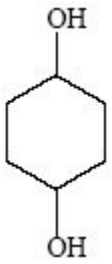
5. Three cyclic alcohols, cyclohexan–1,2-diol, cyclohexan–1,3-diol and cyclohexan–1,4-diol were compared using ^{13}C n.m.r. spectroscopy.



cyclohexan-1,2-diol

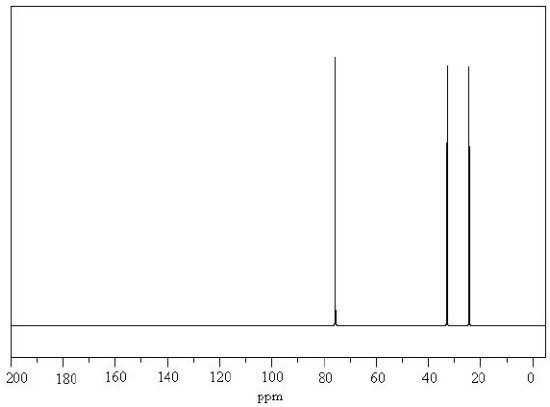


cyclohexan-1,3-diol



cyclohexan-1,4-diol

The ^{13}C n.m.r. spectrum of cyclohexan-1,2-diol is shown below.



(a) (i) Explain why there are three peaks.

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(ii) Proton n.m.r. chemical shift data is shown in Table 1 on the reverse of the Periodic Table. Chemical shift values for ^{13}C vary similarly with chemical environment.

Suggest the δ value of the peak in the spectrum above which corresponds to the absorption for carbon atom 1 in cyclohexan-1,2-diol.

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(b) (i) Predict the number of peaks in the ^{13}C n.m.r. spectrum of cyclohexan-1,3-diol.

.....

(ii) Predict the number of peaks in the ^{13}C n.m.r. spectrum of cyclohexan-1,4-diol.

.....

(c) Suggest why the structures drawn above represents several stereoisomers.

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(Total 5 marks)

6. Each of the parts (a) to (e) below concerns a different pair of isomers.

Draw one possible structure for each of the species **A** to **J**, using Table 2 on the Data Sheet where appropriate.

(a) Compounds **A** and **B** have the molecular formula C_5H_{10}
A decolourises bromine water but **B** does not.

A **B**

(2)

(b) Compounds **C** and **D** have the molecular formula $C_2H_4O_2$

Each has an absorption in its infra-red spectrum at about 1700 cm^{-1} but only **D** has a broad absorption at 3350 cm^{-1}

C **D**

(2)

(c) Compounds **E** and **F** are esters with the molecular formula $C_5H_{10}O_2$

The proton n.m.r. spectrum of **E** consists of two singlets only whereas that of **F** consists of two quartets and two triplets.

E **F**

(2)

(d) Compounds **G** and **H** have the molecular formula $C_3H_6C_{l2}$ **G** shows optical activity but **H** does not.

G **H**

(2)

(e) Compounds **I** and **J** have the molecular formula C_6H_{12}

Each has an absorption in its infra-red spectrum at about 1650 cm^{-1} and neither shows geometrical isomerism. The proton n.m.r. spectrum of **I** consists of a singlet only whereas that of **J** consists of a singlet, a triplet and a quartet.

I **J**

(2)
(Total 10 marks)